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**ANALYSIS OF n-BUTYL
CHLOROPHENOXYACETATES BY GAS
CHROMATOGRAPHY AND INFRARED
SPECTROSCOPY**

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Mellon Institute

TECHNICAL REPORT AFML-TR-69-281

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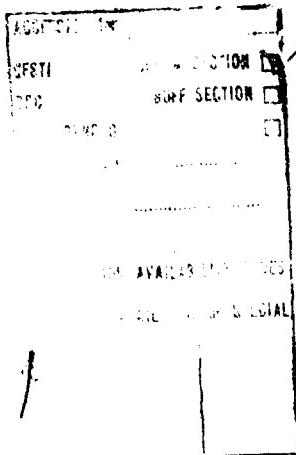
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FOREWORD

This report was prepared by the Analytical Branch, Materials Physics Division, Air Force Materials Laboratory. This work was initiated under Project No. 7360, "The Chemistry and Physics of Materials," Task No. 736005, "Compositional Atomic, and Molecular Analysis," and administered by the Air Force Materials Laboratory, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio, Ronald G. Lewis, Capt., USAF (MAYA), Wm. J. Crawford (MAYA), and Wilbert R. Powell (MAYA). The contractual portion of the work with Mellon Institute (AF 33 (615)-68-C-1369) was directed by Mr. F. F. Bentley.

This report covers work performed from September 1968 to July 1969. The report was submitted by the authors in July 1969.

This technical report has been reviewed and is approved.

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ABSTRACT

A gas chromatographic method was developed for the analysis of samples of n-butyl 2,4-dichlorophenoxyacetate, n-butyl 2,4,5-trichlorophenoxyacetate, and mixtures of these two compounds. These mixtures are called herbicide orange.

Samples of n-butyl 2,4-dichlorophenoxyacetate (four), n-butyl 2,4,5-trichlorophenoxyacetate (four), and herbicide orange (15) were analyzed by gas chromatography. No less than 23 impurity peaks were obtained in the collective chromatograms. Six peaks (designated H, I, J, Q, R, and S on the chromatograms) were the most common impurities. They appeared in a number of chromatograms and often accounted for a major portion of the impurity content. The number and amount of impurities varied considerably in the different samples, even in samples from the same source.

The n-butyl 2,4-dichlorophenoxyacetate and the n-butyl 2,4,5-trichlorophenoxyacetate content of the herbicide orange samples was determined by both infrared and gas chromatographic methods. The results from the two methods are compared, and some of the factors which influenced these results and their reliability are discussed.

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SECTION I
INTRODUCTION

A gas chromatographic (GLC) procedure was developed to determine the purity of n-butyl 2,4-dichlorophenoxyacetate (designated "D") and n-butyl 2,4,5-trichlorophenoxyacetate (designated "T") samples. This method was employed to determine the weight percent of "D" and "T" contained in samples of herbicide orange and other mixtures of "D" and "T" prepared in this laboratory.

The "D" and "T" content of the samples of herbicide orange was determined by an infrared spectroscopic method (Reference 7). The analytical results from the infrared method are compared with the results of the gas chromatographic analyses.

SECTION II

BACKGROUND

Samples of "D" and "T" were obtained from commercial and government sources, and all were required to meet military specifications (References 1, 2). The military specifications required tests for total acid equivalence (Reference 3), free acid (Reference 4), moisture content (Reference 5), and specific gravity (Reference 6). The samples were to contain no less than 95% "D" or "T".

The orange samples were obtained from commercial and government sources, and all were formulated according to military purchase description (Reference 7). However, this purchase description contains a highly restrictive analytical infrared procedure for determining the "D" and "T" content of the orange samples. The validity of applying this analytical infrared test to production samples of orange was questioned.

It was recognized that the nature and quantity of the impurities contained in the "D" and "T" samples used to make herbicide orange could vary greatly from supplier to supplier. Since only the physical properties of the materials are tested in the specifications (References 1, 2) and since these physical properties are not strictly indicative of chemical purity, the impurities contained in the "D" and "T" samples could be considerable and varied. Thus, the nature and quantity of impurities contained in the "D" and "T" components could cause the infrared test for orange quality to be impractical for production materials.

In order to determine if the impurity of the "D" and "T" components could be a major problem, a quantitative determination of the major impurities as well as a determination of how these impurities varied from sample to sample was needed. Thus, a GLC study of "D", "T", and orange samples was undertaken.

SECTION III

EXPERIMENTAL

Samples were obtained from commercial and government sources and are designated as A-G depending upon the source from which they were obtained.

1. GAS CHROMATOGRAPHIC ANALYSIS

Analyses were attempted on a Varian Aerograph 202b gas chromatograph (with 1/4 inch O D columns) and on a F&M 5750 analytical research gas chromatograph (with 1/8 inch O D columns). The following column coatings were tested for application to this problem: DC-550, polysulfone, polyphenyl ether (7-ring), W-98, OV-17, FFAP, isopentyl sulfide, SE-30, and XE-60. A number of isothermal temperatures as well as temperature programs were studied to determine maximum separation and resolution.

The following procedure was chosen as the one giving the best separations and the most reproducible results.

- (a) Instrument: F&M 5750 Analytical Research Chromatograph
- (b) Column: 6 feet. 1/8 inch O D, coating XE-60, 10% by weight, support chromosorb W-AW/DMCS, 80-100 mesh
- (c) Carrier Gas: He, 18 ml/min, inlet pressure 56 PSIG
- (d) Temperatures: Column 150 to 325°C, detector 390°C, injector 260°C, program rate 1.0°C/min.

The samples were diluted with an equal volume of benzene before injection, and a 1.5 to 2.0 μ l sample was injected. The program rate of 1.0°C/min was started immediately after the benzene peak was eluted from the column. The column temperature was held at 235°C, after being reached during the chromatogram, until a total time of two hours was attained after the injection, before the chromatogram was considered complete.

In order to check the accuracy and reproducibility of the GLC Method, four equal volume mixtures of n-butyl 2,4-dichlorophenoxyacetate, Sample B, and n-butyl 2,4,5-trichlorophenoxyacetate, Sample B, were prepared and chromatographed. The mixtures were diluted with an equal volume of benzene before injection. One sample contained 5 ml of each component, a second mixture contained 3 ml of each component, the third mixture contained 2 ml of each component and the fourth mixture contained 1 ml of each component.

2. INFRARED ANALYSIS

The procedure given in the Air Force Purchase Description AFPID 6840-1 (Reference 7) was used for the infrared analysis of herbicide orange samples. The data was recorded using a Perkin-Elmer Model 521 infrared spectrophotometer. To minimize any errors due to solvent, spectro grade carbon disulfide was used in place of the suggested reagent grade solvent.

SECTION IV

RESULTS

Gas chromatograms were recorded for each sample, and several of these chromatograms are represented in Figures 1 to 14. In these chromatograms, the letter N represents n-butyl 2,4,5-trichlorophenoxyacetate and the letter K represents n-butyl 2,4-dichlorophenoxyacetate. The other letters of the alphabet represent impurities found to be present in the samples.

A tabulation of the weight percent composition (Reference 8) of six orange samples is represented in Table I, and a tabulation of the percent composition of each "D" and "T" sample is presented in Table II. The letters of the alphabet representing peaks listed in the tables correspond to the letters assigned to peaks in Figures 1 to 14.

Table III shows the results obtained on the samples prepared to test the accuracy and reproducibility of the GLC method.

Analytical results using both the present infrared method and the new GLC method proposed in this report are shown in Table IV. Fifteen herbicide orange samples were analyzed by both methods, and the total "D" plus "T" content found by both methods is compared in Table IV.

SECTION V

DISCUSSION

In order to determine the best conditions for the GLC separation of the components of herbicide orange, a fairly intensive GLC study of the herbicide was undertaken. A number of different columns were tested for the separation. Column materials such as DC-500, FFAP, and SE-30 were found to afford a fair degree of separation, but a column coating of XE-60 was found to be the best material. Also, as might be expected, 1/8 inch O D columns were found to afford better separations than 1/4 inch O D columns.

Attempts to separate the materials under isothermal column conditions were only partly successful. Under these conditions "D" and "T" could be separated from each other, but separation of impurities peaks from the "D" and "T" peaks was found to be very poor. A programmed temperature increase was found to afford much better separation. The program rate determines the time required for the analysis. A rate of one degree/minute was chosen for these studies as a good compromise between total time needed for the analysis and peak separation.

The chromatograms (Figures 1 to 14) and the results shown in Table I indicate that a fairly large number of impurities exist in most of the herbicide orange samples. The results shown in Table II indicate that these impurities are mainly due to impure "D" and "T" individual components that are used in the formulation of herbicide orange.

Table I and II also indicate that the qualitative and quantitative nature of the impurities vary considerably from sample to sample, even though the samples may be from the same supplier. Thus, it can be concluded that the process(s) for the synthesis of these components and their purification is not a very accurate and reproducible one. Thus, the quantity and nature of the impurities cannot be accurately predicted and eliminated before herbicide orange formulation.

The authors had not used the infrared method before, but during this investigation, discovered several areas of concern that will not be encountered when using the GLC method.

First, carbon disulfide has a high vapor pressure and extreme care must be taken in preparing the calibration plots. Especially with "T" because the material tends to precipitate from solution as the solvent rapidly evaporates.

Secondly, the infrared method specifies a compensating solution of "D" in the reference beam, when measuring the "T" concentration. The article states the concentration of "D" should be equal to the amount of "D" already determined in the sample. Then, in parenthesis it suggests 45 to 50 mg/ml of "D" in carbon disulfide. The authors found that unless the actual "D" concentration is used, the value for the concentration of "T" will be in error by 2 to 4% depending on what concentration of "D" is chosen.

Thirdly, Table IV shows the IR method generally gives results higher than the GLC method. The impurities in the various suppliers samples can enhance the infrared results. This is especially noticeable in Sample G, where the infrared analysis for "T" gave 34.77%, but the GLC method gave only 1.82%. The authors found that Sample G had at least three impurities, which apparently gave rise to an absorption band at 1160 cm^{-1} . The infrared analysis would only indicate a bad batch of orange herbicide while the GLC reveals the orange herbicide virtually lacks one important component.

Attempts to identify several of the major impurities that were found to be present in most of the samples by combined gas chromatography-mass spectral analysis have to date not been successful. However, Sample G in Table IV is an excellent case where the impurities in the sample afford infrared peaks in the regions of interest. This should be expected since the peaks of interest are not specific for "D" and "T". Thus, it is to be expected that compounds with a composition similar to "D" or "T" could afford infrared peaks in the regions of interest.

REFERENCES

1. Military Specification, MIL-H-51147A (MU), November 7, 1968.
2. Military Specification, MIL-H-51148A (MU), November 7, 1968.
3. Official Methods of Analysis of the Association of Official Agricultural Chemists, 9th ed., paragraphs 4.130, 4.131, 4.132, 4.161(a) or 4.161(c), 1960.
4. American Society for Testing and Materials, ASTM Standards, D664.
5. American Society for Testing and Materials, ASTM Standards, D1533.
6. American Society for Testing and Materials, ASTM Standards, D70.
7. Purchase Description, AFPID 6840-1, Amendment 1, April 11, 1968.
8. Leslie S. Ettre and Albert Zlatkis, The Practice of Gas Chromatography, New York, Interscience Publishers, 1967.

GAS CHROMATOGRAMS

The graphs show the gas chromatograms of "D", "T", and orange herbicide samples. The letter designation for each sample refers to the supplier of the material. The letters assigned to various peaks on the graphs represent components detected. Also shown are the quantities of the major components. The authors attempted to assign the peaks in alphabetical order. However, a few peaks were discovered in samples from companies not initially analyzed (samples obtained near the completion of the research). These peaks were assigned irrespective of alphabetical order to identify all peaks.

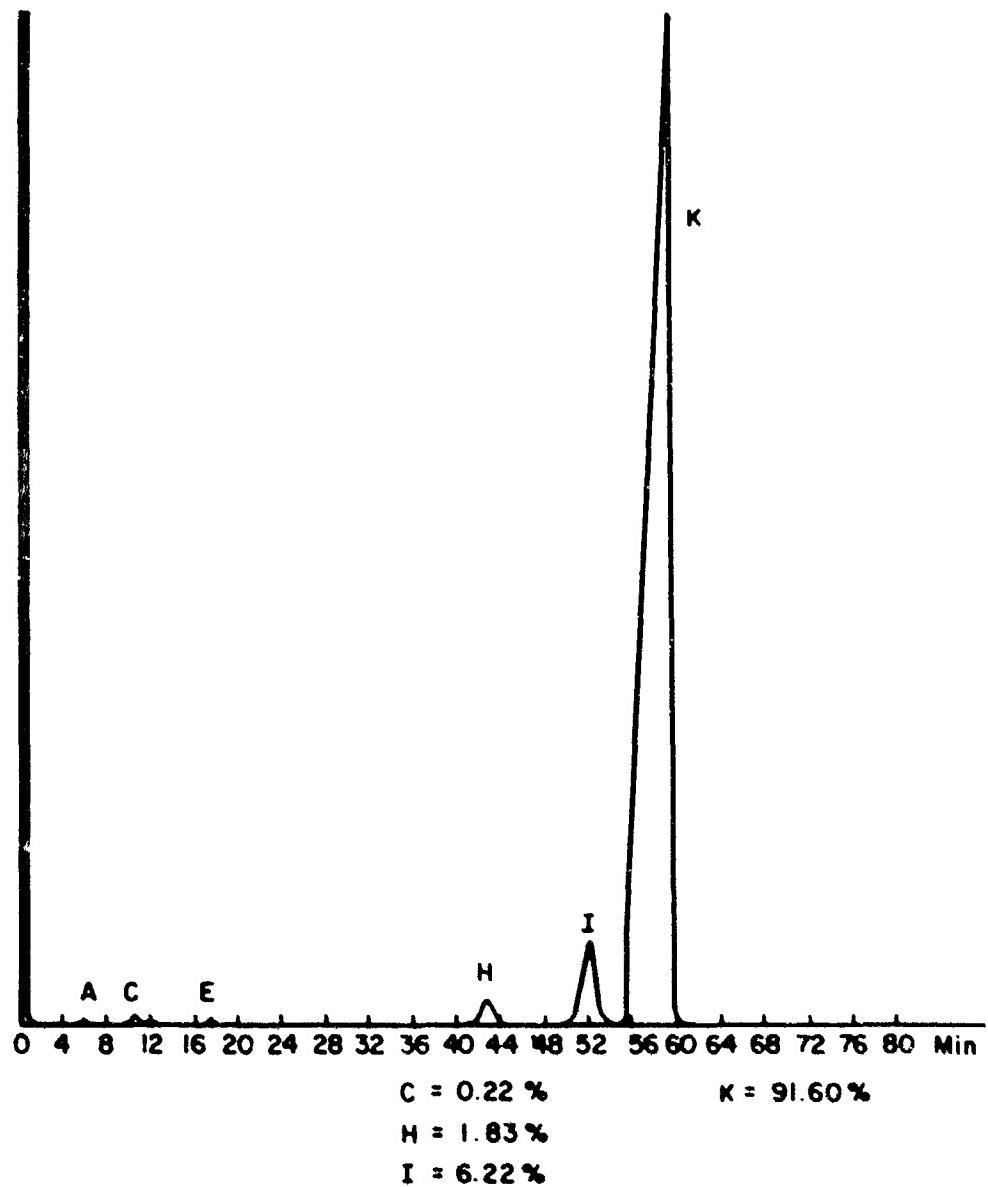


Figure 1. Gas Chromatogram of n-Butyl 2,4-Dichlorophenoxyacetate Sample A

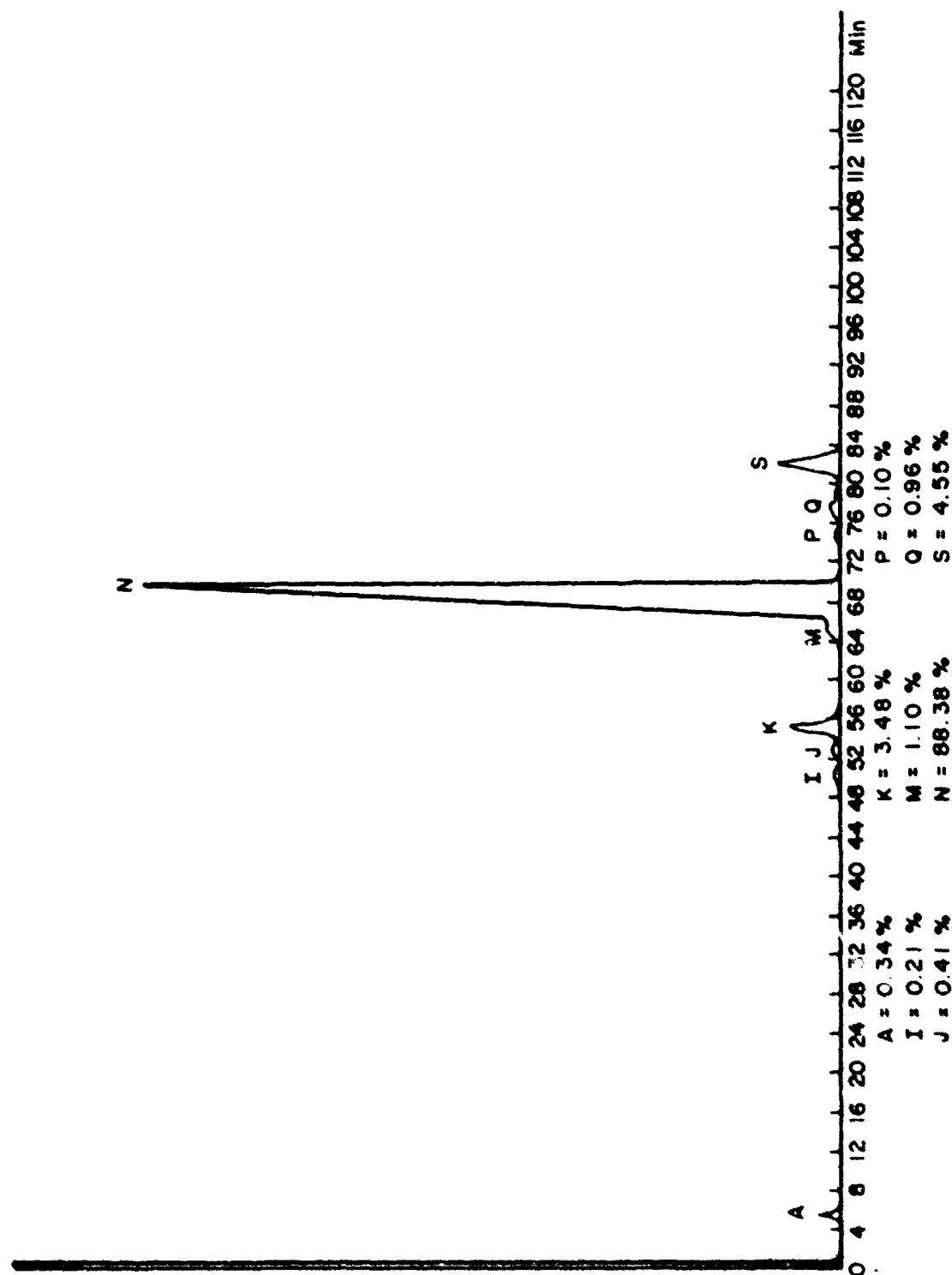


Figure 2. Gas Chromatogram of n-Butyl 2,4,5-Trichlorophenoxyacetate Sample A

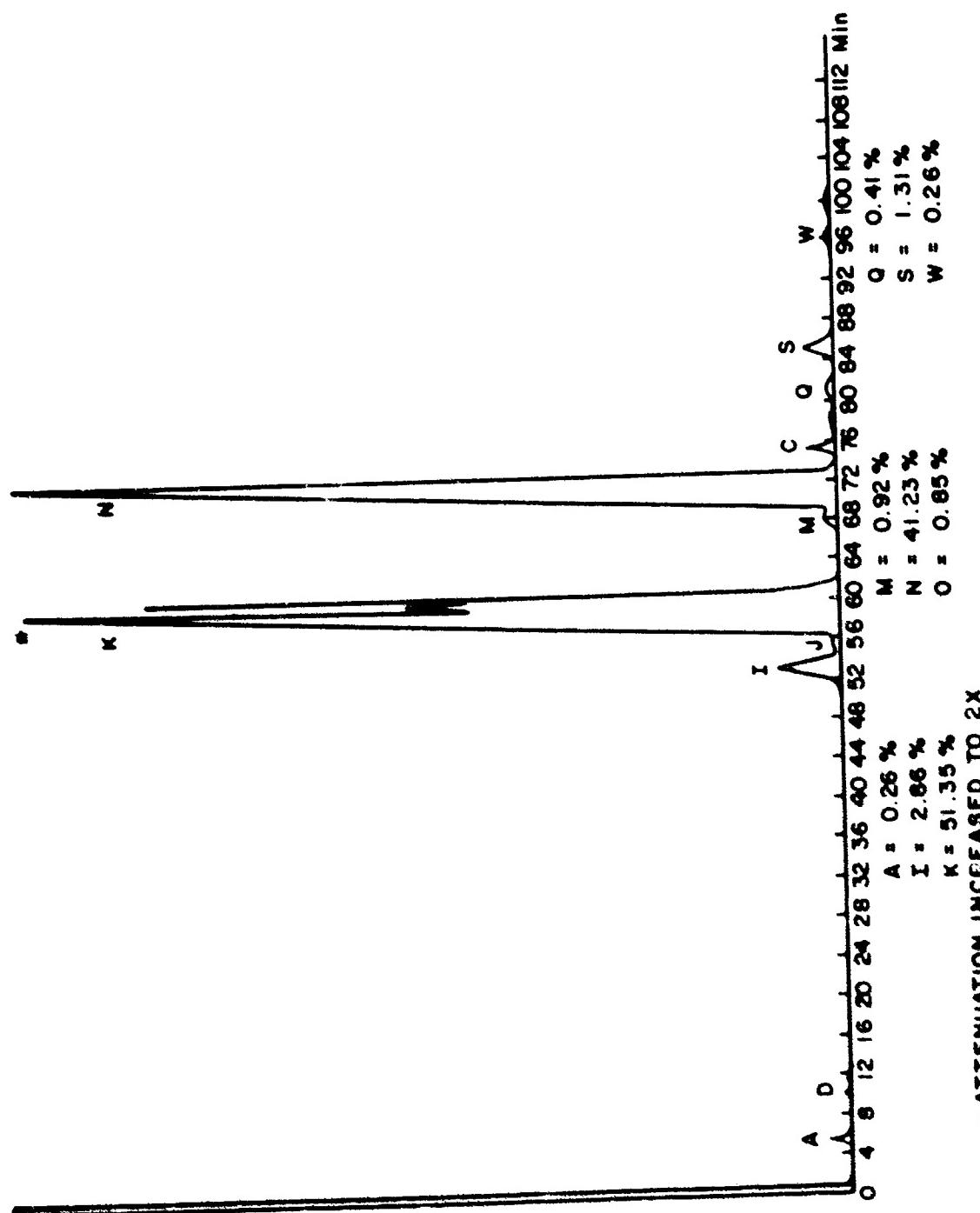


Figure 3. Gas Chromatogram of Herbicide Orange Sample A
n-Butyl 2,4-Dichlorophenoxyacetate and n-Butyl 2,4,5-Trichlorophenoxyacetate

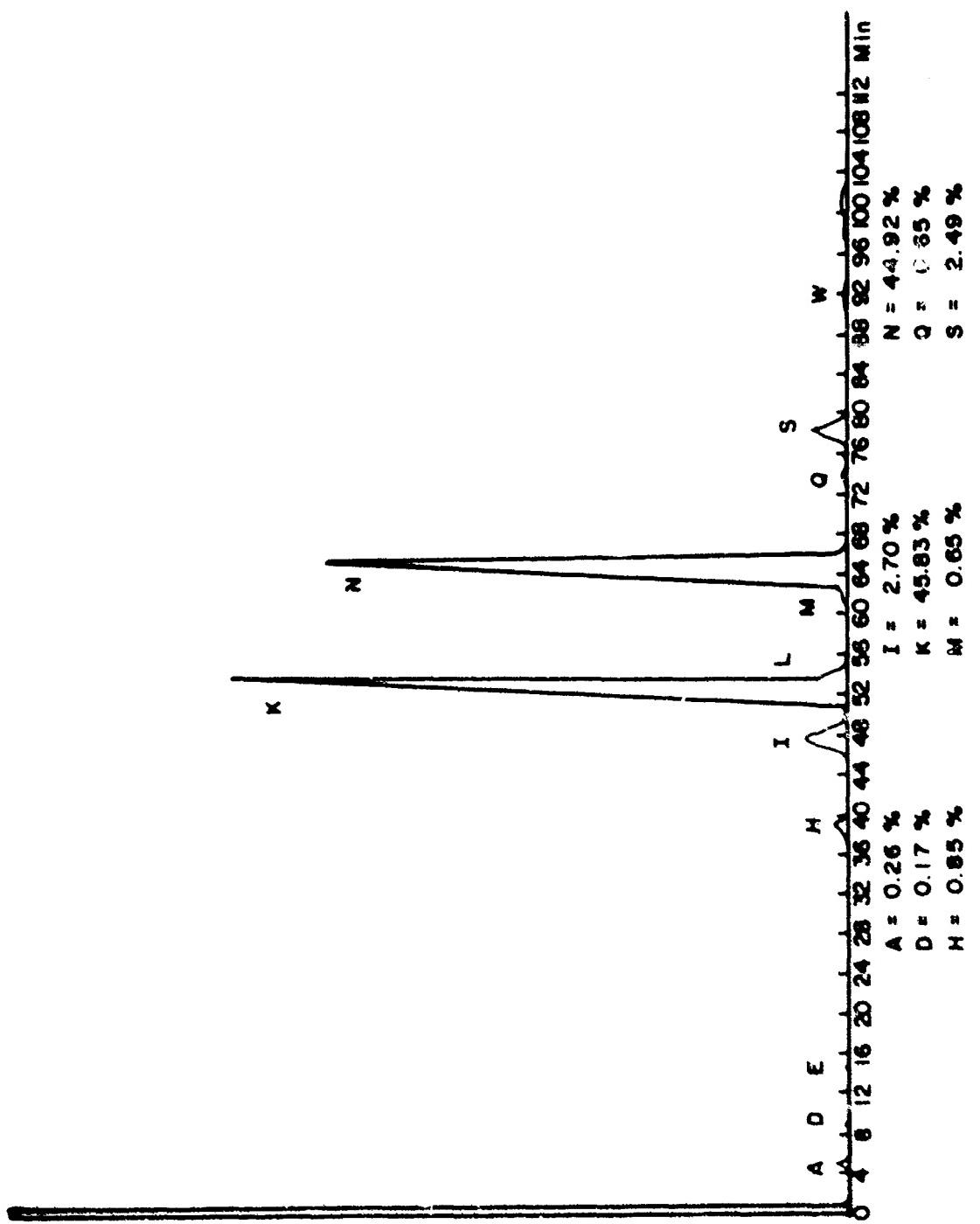


Figure 4. Gas Chromatogram of Herbicide Orange Sample A₁
n-Butyl 2,4-Dichlorophenoxyacetate and n-Butyl 2,4,5-Trichlorophenoxyacetate

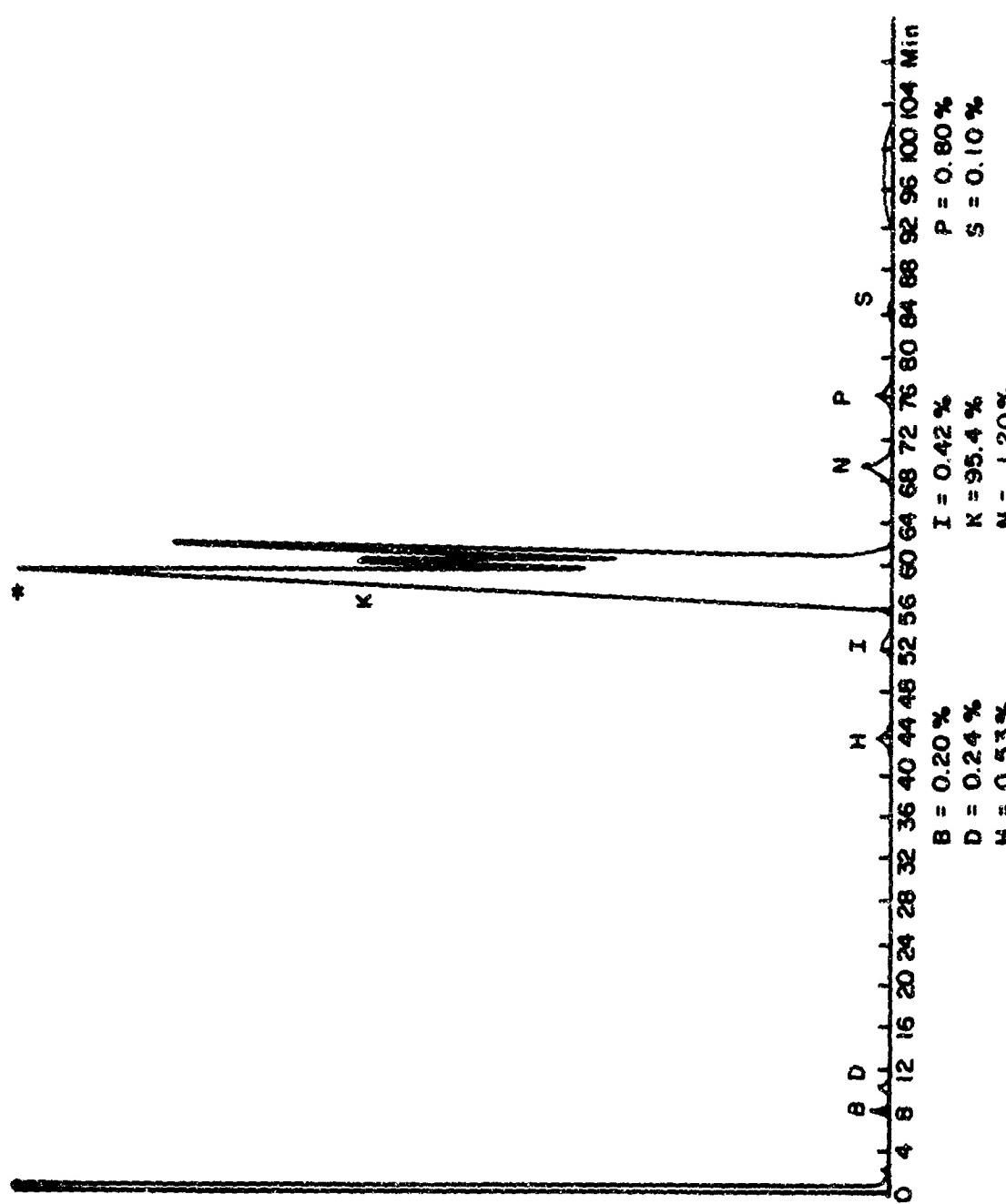


Figure 5. Gas Chromatogram of n-Butyl 2,4-Dichlorophenoxyacetate Sample B

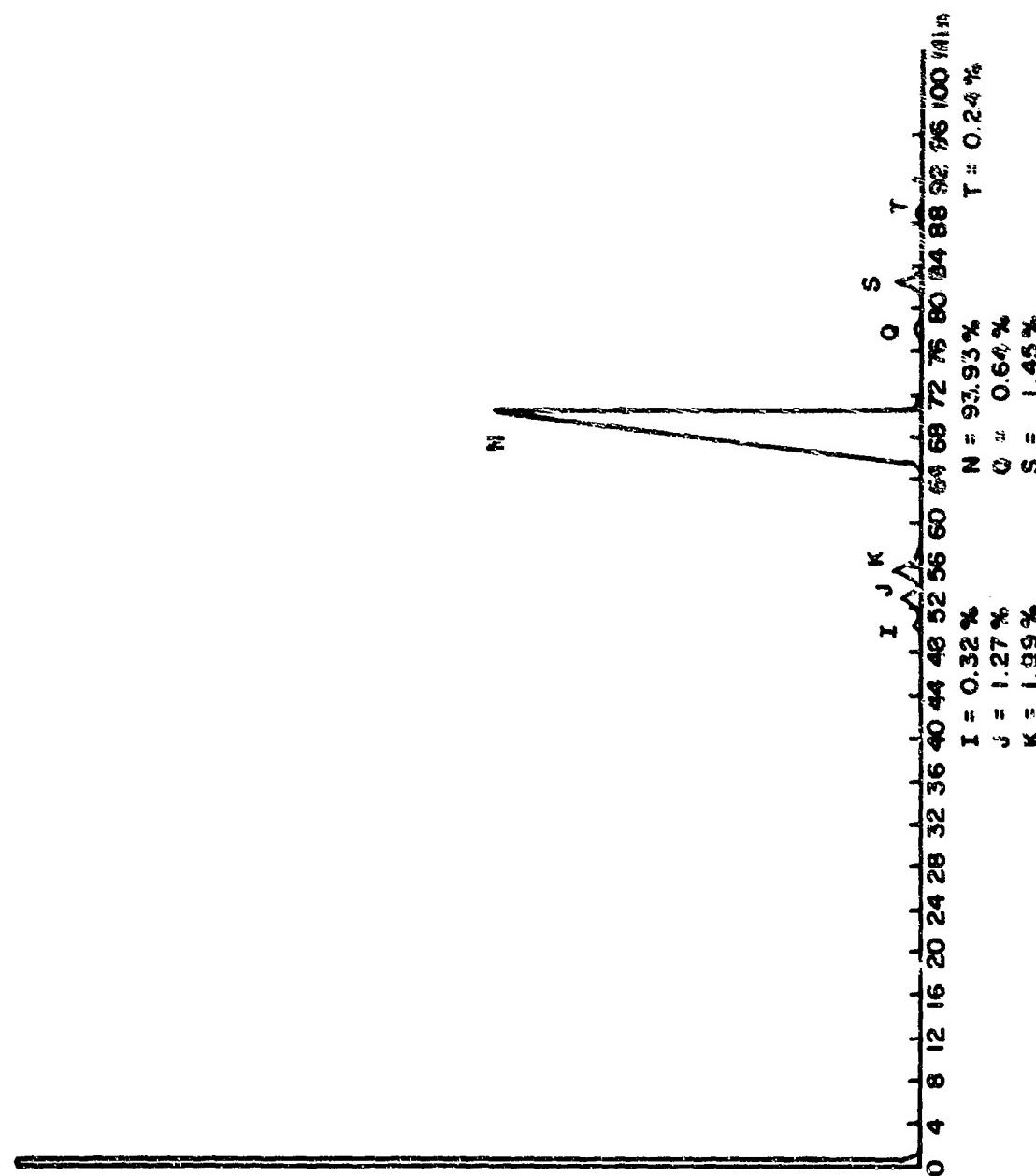


Figure 6. Gas Chromatogram of n-Butyl 2,4,5-Trichlorophenoxyacetate, Sample B

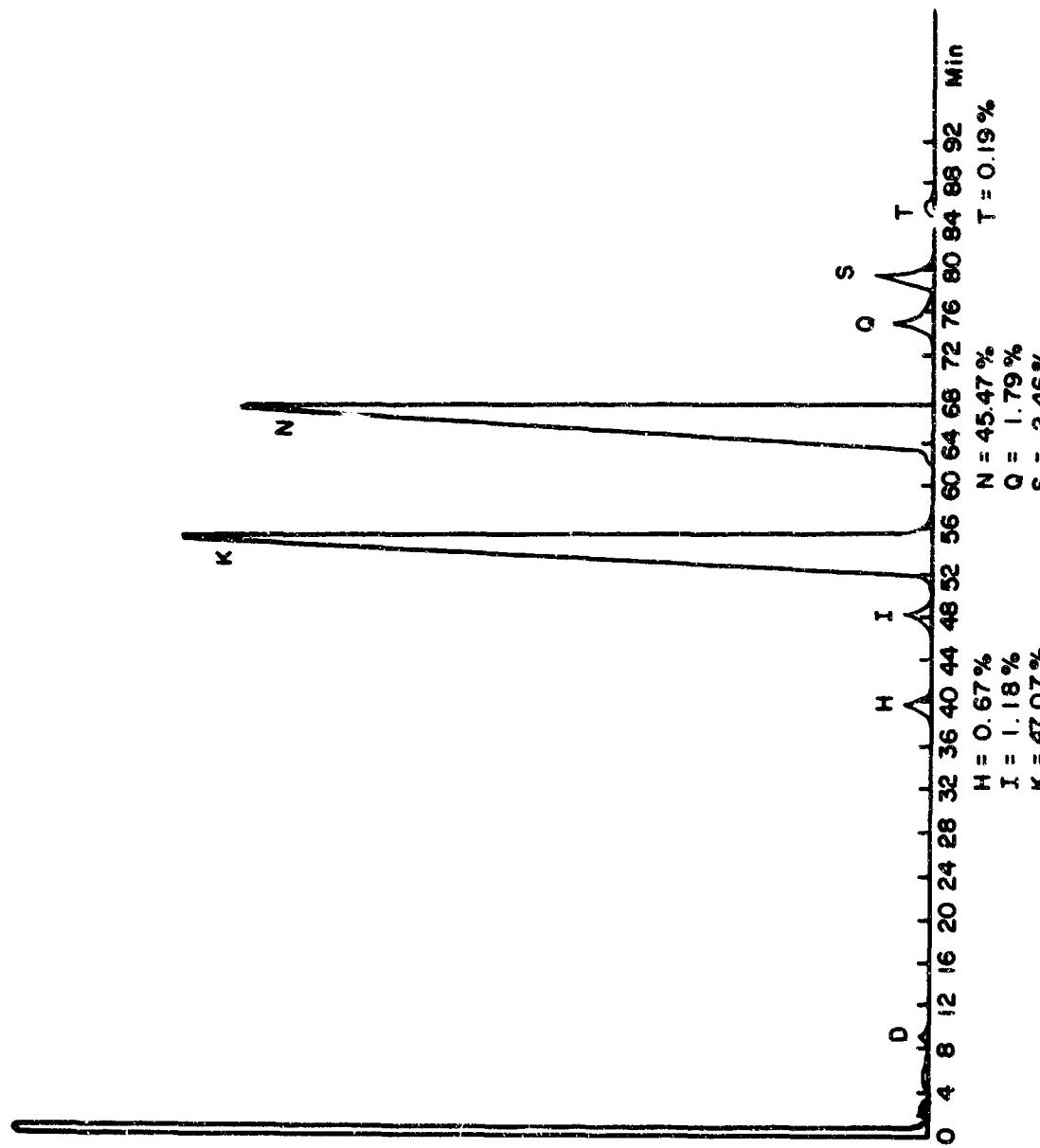


Figure 7. Gas Chromatogram of Herbicide Orange Sample B
n-Butyl 2,4-Dichlorophenoxyacetate and n-Butyl 2,4,5-Trichlorophenoxyacetate

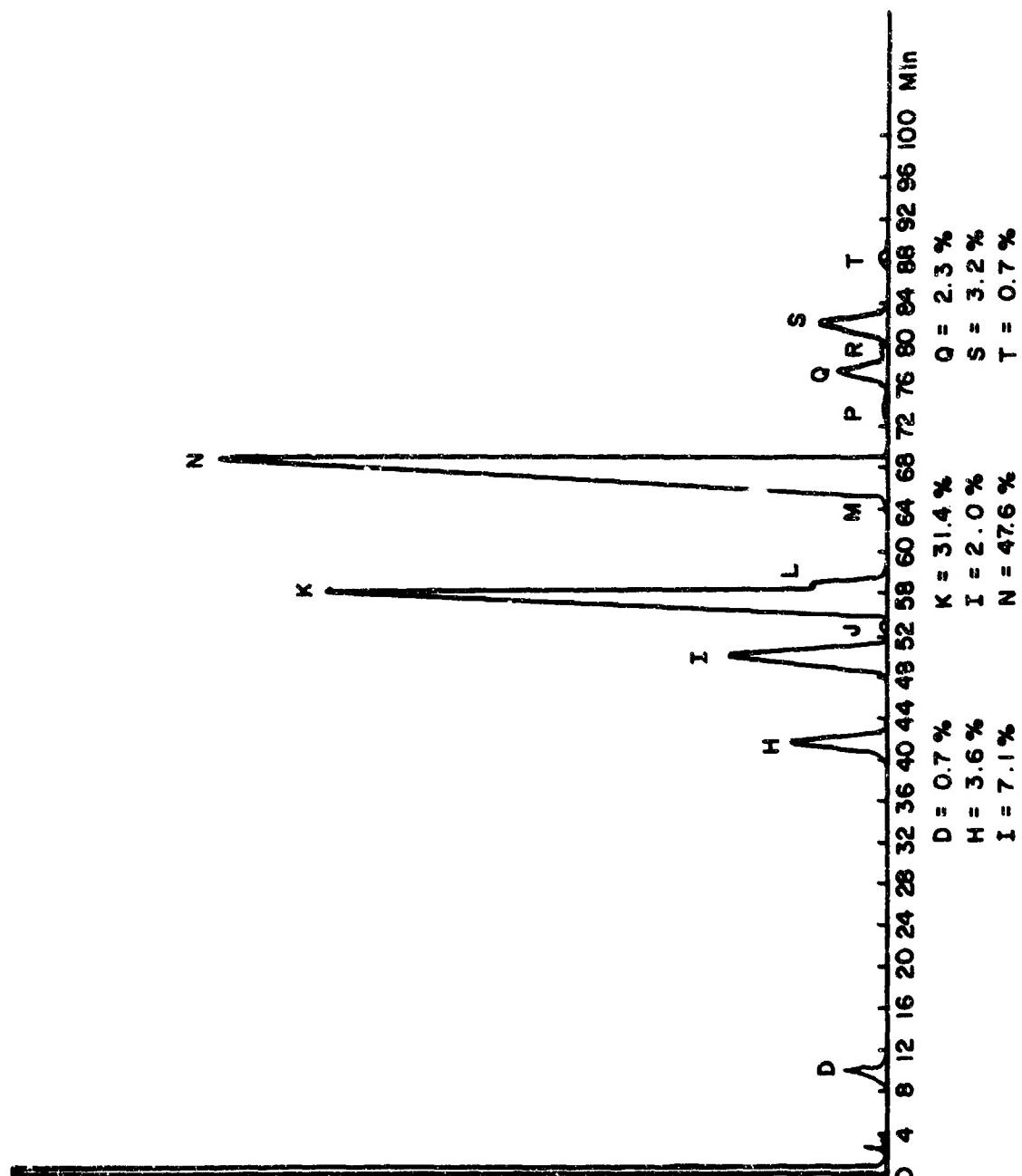


Figure 8. Gas Chromatogram of Herbicide Orange Sample B₁
n-Butyl 2,4-Dichlorophenoxyacetate and n-Butyl 2,4,5-Trichlorophenoxyacetate

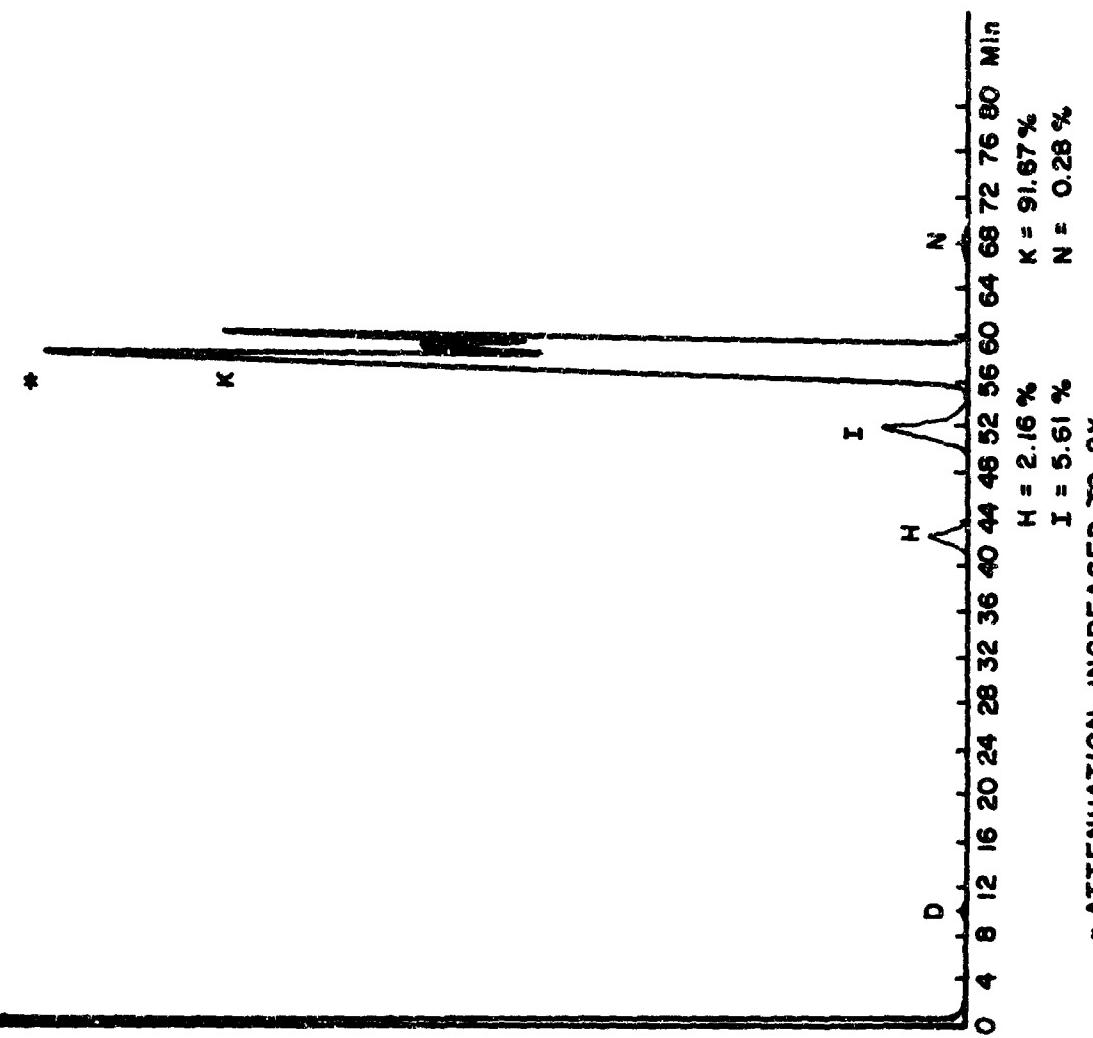


Figure 9. Gas Chromatogram of n-Butyl 2,4-Dichlorophenoxyacetate Sample C

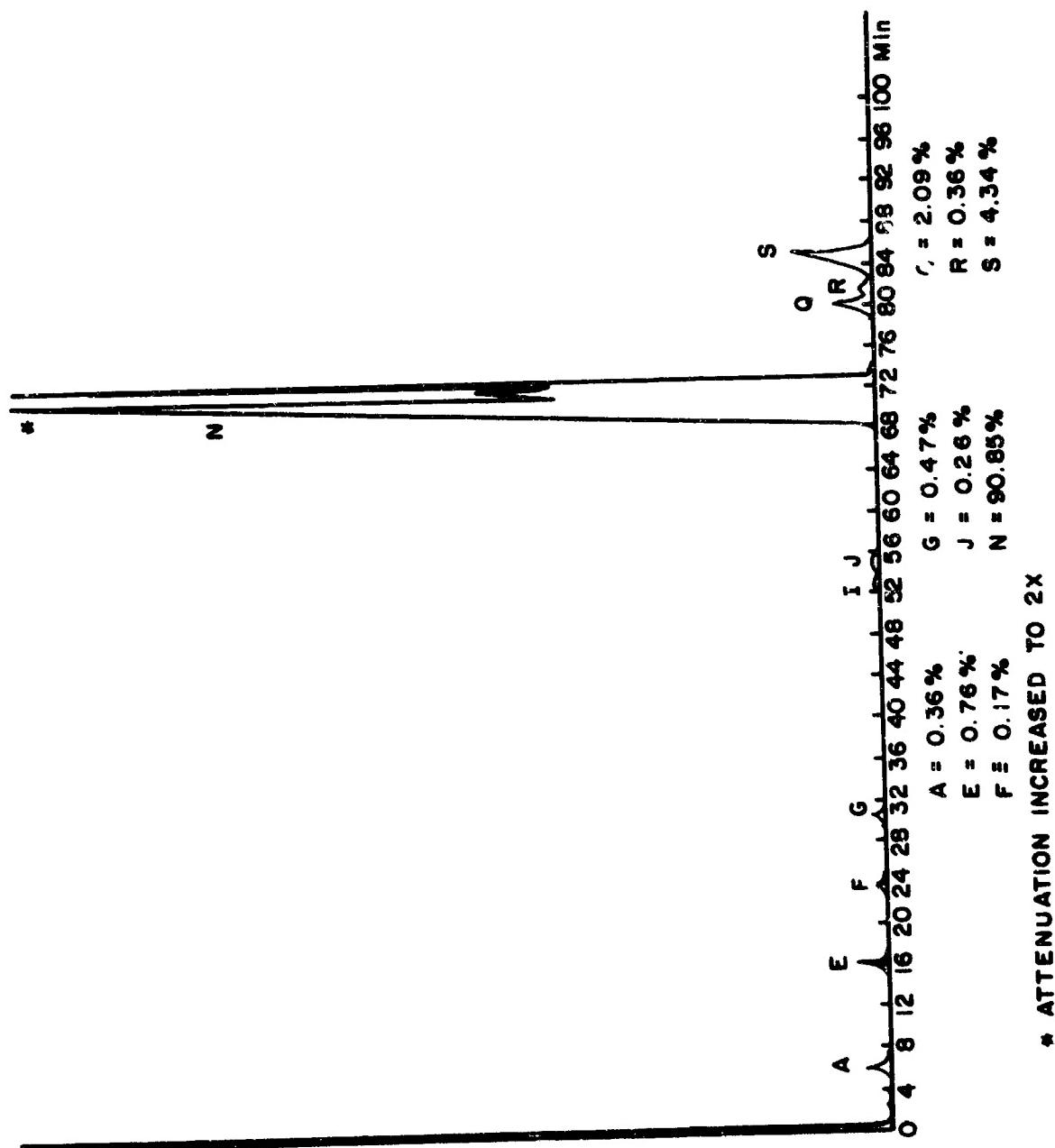


Figure 10. Gas Chromatogram of n-Butyl 2,4,5-Trichlorophenoxyacetate Sample C

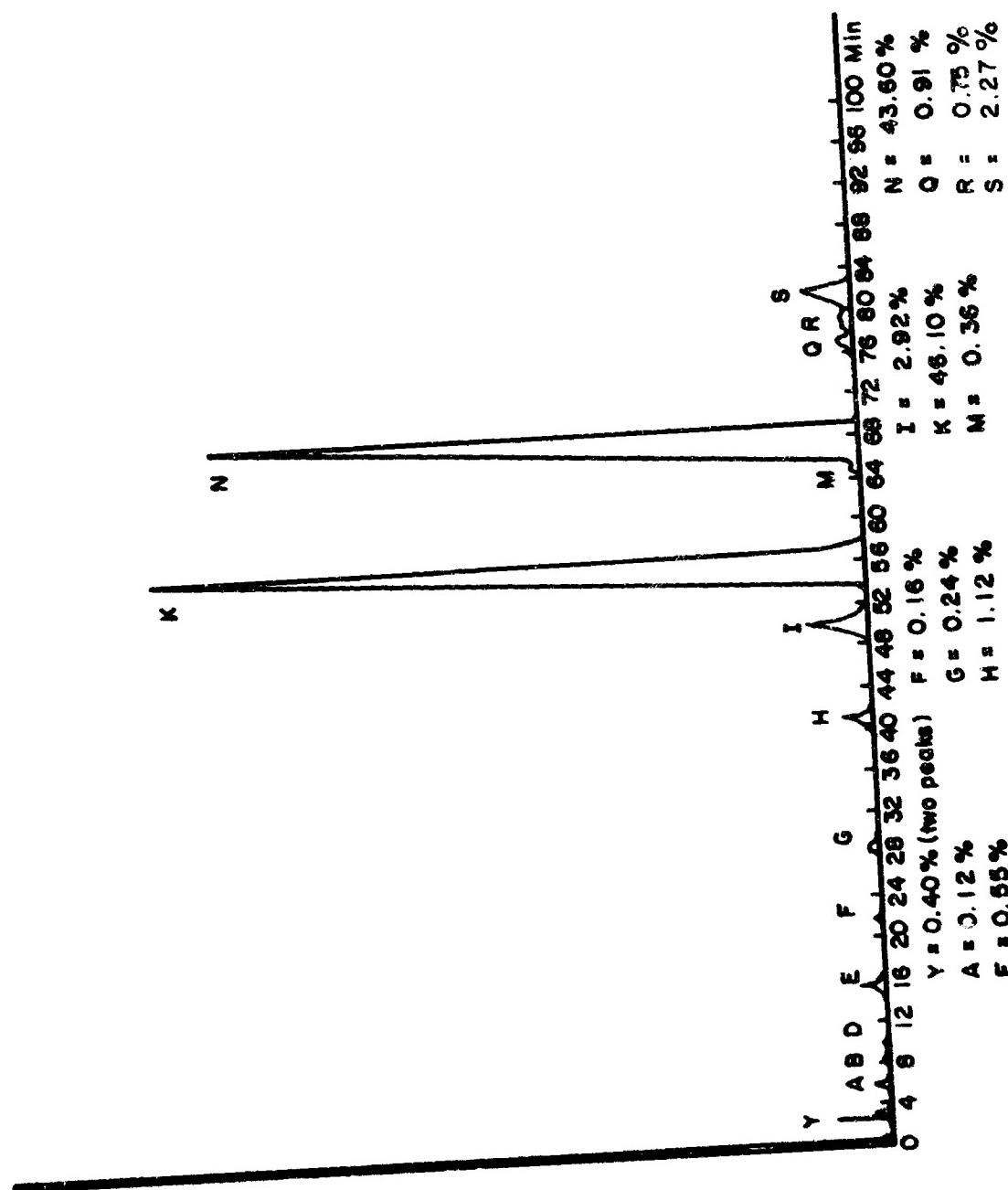


Figure 11. Gas Chromatogram of Herbicide Orange Sample C
n-Butyl 2,4-Dichlorophenoxyacetate and n-Butyl 2,4,5-Trichlorophenoxyacetate

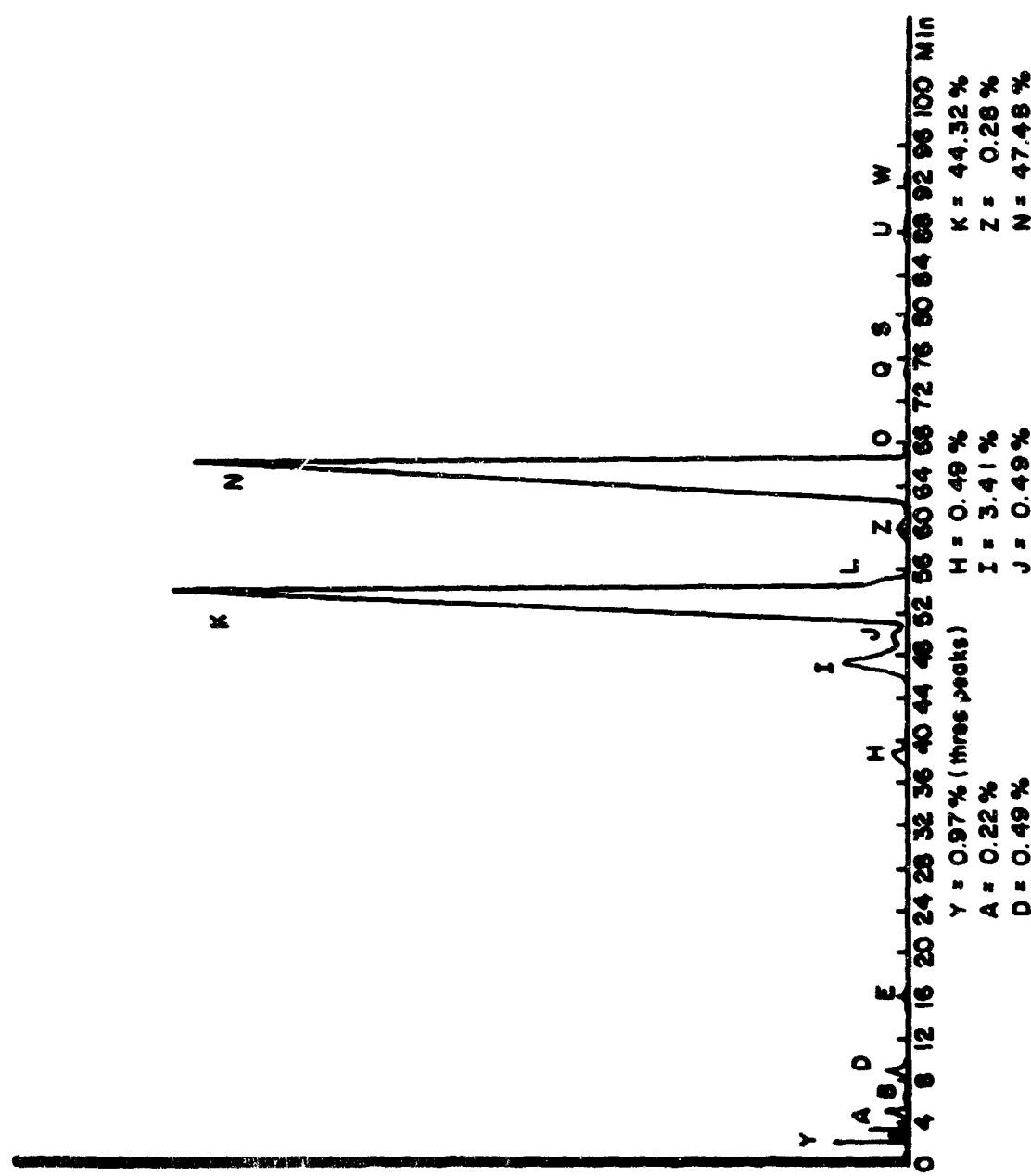


Figure 12. Gas Chromatogram of Herbicide Orange Sample D
n-Butyl 2,4-Dichlorophenoxyacetate and n-Butyl 2,4,5-Trichlorophenoxyacetate

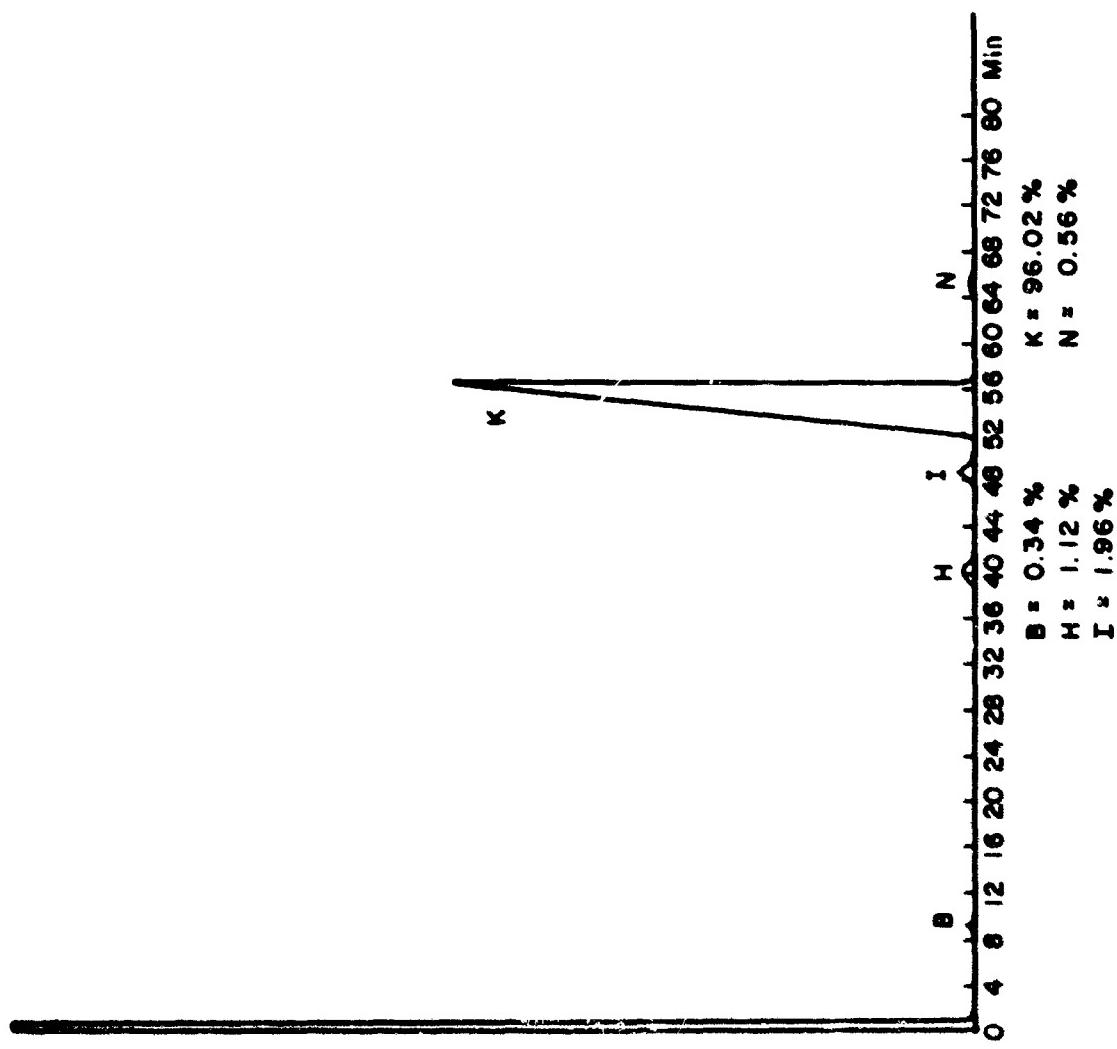


Figure 13. Gas Chromatogram of n-Butyl 2,4-Dichlorophenoxyacetate Sample E

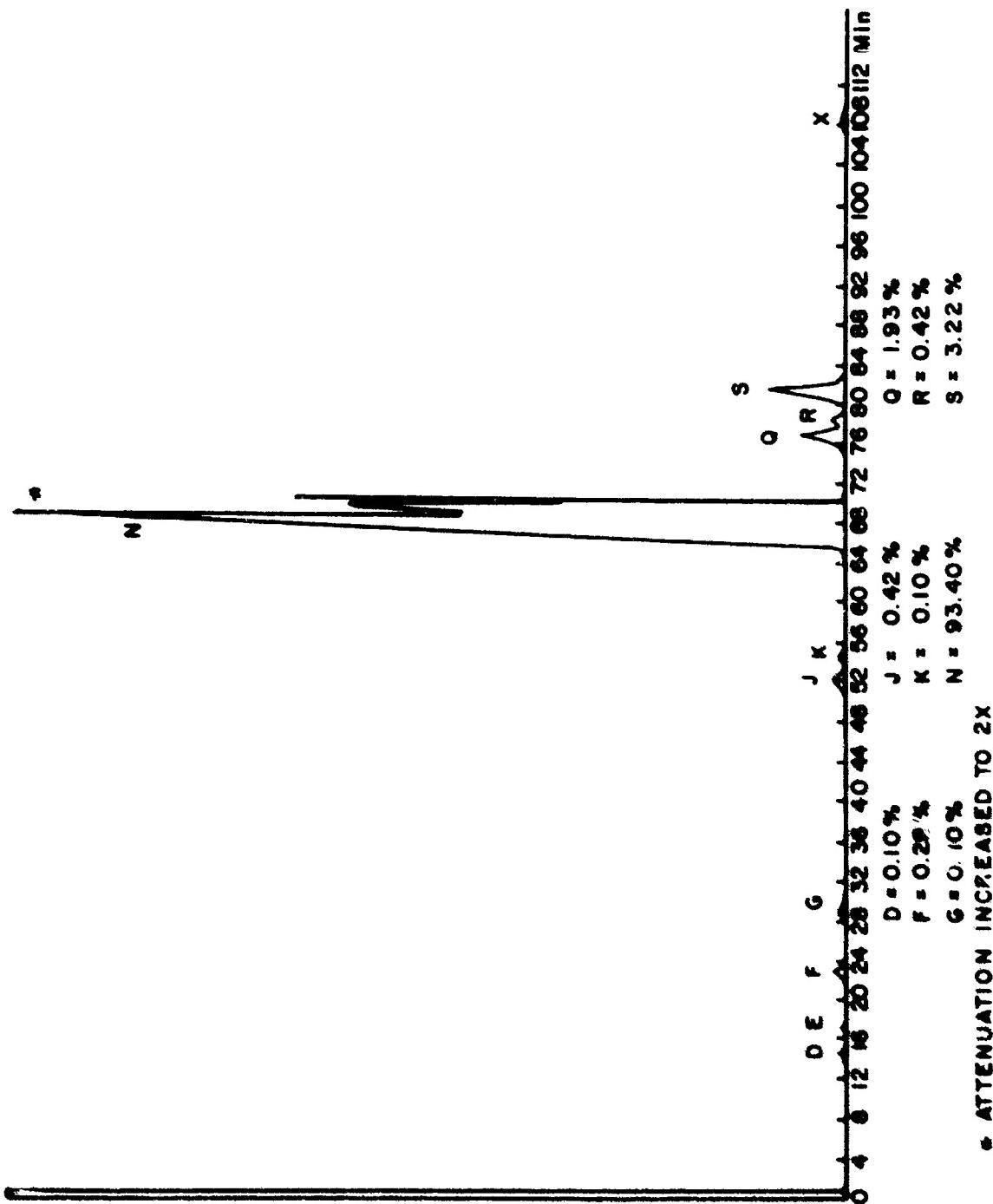


Figure 14. Gas Chromatogram of n-Butyl 2,4,5-Trichlorophenoxyacetate Sample E

TABLES

The authors have compiled tables to show the analytical data for "D", "T", and orange herbicide samples. Due to trace amounts of some components it was convenient to combine these components into columns Y-E and T-X. The columns Y through E and T through X represent components Y, A, B, C, D, E, and T, U, V, W, X, respectively.

TABLE I
GAS CHROMATOGRAPHIC ANALYSIS OF HERBICIDE ORANGE SAMPLES

Sample	PEAK (% by weight)																
	Y-E	F	G	H	I	J	K*	L	Z	M	N**	O	P	Q	R	S	T-X
A	.3	No	No	T*	2.9	.3	51.4	?b	No	.9	41.2	.9	T	.4	T	1.3	.5
A ₁	.4	T	No	.9	2.7	.2	46.8	.9	N	.7	44.9	No	?	.7	?	2.5	.4
B	.3	No	?	.7	1.2	T	47.1	.6	No	.1	45.5	No	?	1.8	.2	2.5	.2
B ₁	.9	No	No	3.6	7.1	.4	31.4	2.0	No	.2	47.6	No	.2	2.3	.5	3.1	.7
C	1.1	.2	.2	1.1	2.9	.1	46.1	?	No	.4	43.6	?	T	.9	.8	2.3	T
D	1.9	No	No	.6	3.4	.5	44.3	1.2	.3	?	47.5	?	No	T	T	.1	.5

a) T = Trace; Peak definitely present but amount too small for quantitative determination
 b) ? = Presence questionable

* n-Butyl 2,4-dichlorophenoxyacetate

** n-Butyl 2,4,5-trichlorophenoxyacetate

TABLE II
GAS CHROMATOGRAPHIC ANALYSIS OF COMPONENTS

Sample	PEAKS (% by weight)															
	Y-E	F	G	H	I	J	K*	L	Z	M	N**	O	P	Q	R	S
n-Butyl 2,4-dichlorophenoxyacetate																
A	.3	No	No	1.8	6.2	No	91.6	? ^b	No	?	No	No	No	No	No	No
B	.4	No	No	.5	.4	?	95.4	?	No	?	1.2	No	No	.8	No	.1
C	T ^a	No	No	2.2	5.6	T	91.7	?	No	?	3	No	No	No	No	No
E	.3	No	No	1.1	2.0	No	96.0	?	No	?	No	No	No	No	No	No
n-Butyl 2,4,5-trichlorophenoxyacetate																
A	.3	No	No	.2	.4		3.5	?		1.1	88.4	No	.1	1.0	T	4.6
B	T	No	No	.3	1.3		2.0	T		?	93.9	No	No	.6	?	1.5
C	1.1	.2	.5	No	.1		T	?	No	?	80.9	No	No	2.1	.4	4.4
E	.1	.3	.1	No	No		.4	.1	No	No	?	93.4	No	No	1.9	.4

a) T = Trace

b) ? = Presence questionable

* K = n-Butyl 2,4-dichlorophenoxyacetate

** N = n-Butyl 2,4,5-trichlorophenoxyacetate

TABLE III***
 GAS CHROMATOGRAPHIC ANALYSIS OF 1:1 (VOLUME) MIXTURES
 OF THE INDIVIDUAL COMPONENTS

Sample	PEAKS (% by weight)																
	Y-E	F	G	H	I	J	K*	L	Z	M	N**	O	P	Q	R	S	T-X
5	.2	No	No	.3	.3	.6	48.0	? ^a	No	?	47.8	No	No	.9	.1	.7	.3
3	.2	No	No	.3	.3	.4	48.5	?	No	?	48.2	No	No	.9	T ^b	.7	.4
2	.2	No	No	.3	.3	.3	48.8	?	No	?	48.1	No	No	.5	T	.7	.2
1	.2	No	No	.3	.3	.3	48.6	?	No	?	48.2	No	No	.8	T	.7	.3

a) ? = Presence questionable

b) T = Trace

* K = n-Butyl 2,4-dichloropropenoxyacetate

** N = n-Butyl 2,4,5-trichloropropenoxyacetate

*** Equal volume mixtures of the components from source Y to check accuracy and reproducibility of the method

5 is a 5 ml -5 ml mixture
 3 is a 3 ml -3 ml mixture
 2 is a 2 ml -2 ml mixture
 1 is a 1 ml -1 ml mixture

TABLE IV
INFRARED VERSUS GAS CHROMATOGRAPHIC ANALYTICAL RESULTS
ON HERBICIDE ORANGE SAMPLES

Sample	IR			GLC		
	%D*	%I*	% (D&T)	%D*	%T*	% (D&T)
A	49.82	45.43	95.2	51.4	41.3	92.6
A ₁	46.98	48.39	95.4	45.8	44.9	90.7
A ₂	46.13	47.73	93.9	45.5	46.9	92.4
A ₃	46.99	48.48	95.5	46.8	47.6	94.4
A ₄	48.40	48.99	97.4	44.9	48.2	93.1
B	46.59	47.39	94.0	47.1	45.5	92.6
B ₁	30.32	47.68	78.0	31.4	47.6	79.0
B ₂	46.52	49.49	96.0	50.4	46.1	96.5
B ₃	45.06	47.84	92.9	47.6	45.9	93.5
C	47.17	46.57	93.7	46.1	43.6	89.7
D	45.47	49.86	95.3	44.3	47.5	91.8
E	47.81	48.31	96.1	50.4	46.1	96.5
E ₁	50.30	45.82	96.1	50.7	45.6	96.3
F	48.25	48.29	96.5	45.9	49.5	95.4
G	48.46	34.77	83.2	50.1	1.8	51.9

*Values are % by weight

APPENDIX

After this investigation had been completed and the report submitted for publication, it was made known to the authors that the "T" content of sample G was not present as the n-butyl ester but rather as an isoocetyl ester. The presence of the isoocetyl ester accounts for the discrepancy in the IR and GLC results. The infrared results were obtained because the absorption band measured was due to a molecular vibration that did not involve the alkyl group in the ester. The value was low and incorrect because the n-butyl ester was used to plot the calibration curve. To accurately determine the "T" content in sample G, a calibration curve should be plotted using the isoocetyl ester.

The presence of "T" as the isoocetyl ester in sample G could also partly explain the large amount of long R_f material found in the chromatogram of sample G. However, definite identification of isoocetyl "T" necessitates further isolation and identification procedures or obtaining a standard sample of the material from the commercial source.

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13. ABSTRACT <p>A gas chromatographic method was developed for the analysis of samples of n-butyl 2,4-dichlorophenoxyacetate, n-butyl 2,4,5-trichlorophenoxyacetate, and mixtures of these two compounds. These mixtures are called herbicide orange.</p> <p>Samples of n-butyl 2,4-dichlorophenoxyacetate (four), n-butyl 2,4,5-trichlorophenoxyacetate (four), and herbicide orange (15) were analyzed by gas chromatography. No less than 23 impurity peaks were obtained in the collective chromatograms. Six peaks (designated H, I, J, Q, R, and S on the chromatograms) were the most common impurities. They appeared in a number of chromatograms and often accounted for a major portion of the impurity content. The number and amount of impurities varied considerably in the different samples, even in samples from the same source.</p> <p>The n-butyl 2,4-dichlorophenoxyacetate and the n-butyl 2,4,5-trichlorophenoxyacetate content of the herbicide orange samples was determined by both infrared and gas chromatographic methods. The results from the two methods are compared, and some of the factors which influenced these results and their reliability are discussed.</p>		

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